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## IN THE UNITED STATES PATENT & TRADEMARK OFFICE

IN RE APPLICATION OF

:

OLEG STENZEL, ET AL.

: EXAMINER: HANOR, SERENA L.

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:

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PRECIPITATED SILICA

## DECLARATION UNDER 37 C.F.R. § 1.132

COMMISSIONER FOR PATENTS ALEXANDRIA, VIRGINIA 22313

SIR:

- I. André Wehmeier , declare and state as follows:
- 1. I am a graduate of FH Muenster and received my diploma degree in the year of 1998. I have been employed by

Evonik Degussa GmbH for 10 years as a chemical

- graduated engineer in the field of product development silica.
- I am familiar with the claims, and have read the Office Action mailed April 18,
   2008, in the above-identified application.
- 3. The following experiments, named Example A, were conducted under my supervision and/or control. In Example A, precipitated silicas according to Examples 4 and 5

of U.S. Patent 6,180,876 (<u>Uhrlandt et al</u>) were compared to precipitated silicas according to Example 1 of the specification of the above-identified application.

## 4. General performance standard operating procedure (SOP):

The inventive silica according to Example 1 of the above-identified application was tested in a typical motor-truck tire compound. Moreover, this formula is also relevant for the carcass of all rubber tires.

The formula used for the rubber mixtures is specified in the following Table A. The unit "phr" therein denotes parts by weight relative to 100 parts of the raw rubber used.

Application No. 10/523,029 Declaration Under 37 C.F.R. § 1.132

Table A: Compound

Amsperger Chemikalien GmbII; 50858 Cologne; Germany Rhein Chemie Rheinau GmbH; 68219 Mannheim Rheinau; Germany Rhein Chemie Rheinau GmbH; 68219 Mannheim Rheinau; Rhein Chemic Rheinau GmbH; 68219 Mannheim Rheinau; Rhein Chemie Rheinau GmbH; 68219 Mannheim Rheinau; Degussa AG; Frankfurt am Main; Germany Caldic Deutschland GmbH & Co. KG; 40231 Düsseldorf; Germany Chemetall GmbH; 60487 Frankfurt a. Main; Germany Degussa AG; Frankfurt am Main; Germany Degussa AG; Frankfurt am Main; Germany Merck KGaA; 64271 Darmstadt; Germany Company Germany Germany Germany Dicyclohexylbenzothiazole sulfonamide (DCBS) Palmitic-stearic acid; "iodine number 1" stearin Finely divided sulfur according to Ph Eur, BP Si 69 (bis(3-triethoxysilylpropyl)tetrasulfide) 80% polymer bound of butylbenzothiazole sulfonamide (TBBS) N-(1,3-Dimethylbutyl)-N'-phenyl-p-Substance Trimethyldihydroquinoline phenylenediamine (6PPD) 10.0 Aromatic plasticizer oil 100 Natural rubber 10 Carbon black Carbon black Lasting / remill step Finish mixing Basic mixing ZPO 3.0 2 2 0.00 1.4 phr 2.0 1.7 0.3 SMR 10 (degraded to MLA = 60 Article designation Rhenogran TBBS-80 ZnO; RS RAL 844 C Vulkanox 4020 / LG Vulkanox HS/LG EDENOR STI GS Vulkacit DZ/EG-C CORAX N 121 CORAX N 121 Ground sulfur Naftolen ZD Step I batch Step 2 batch Silica (KS) 2ªd step 3rd step Formula to 70) Si 69

5. The general method for manufacture of rubber mixtures and their vulcanized derivatives is described in the following book: "Rubber Technology Handbook", W. Hofmann, Hanser Verlag 1994. The specific mixing conditions for the various compounds are presented in Table B.

Table B: Mixing SOP

Mixing SOP				
1" step	W&P GK 1.5E kneader, filling level 0.56,			
	80 rpm, flow temperature 90 °C,			
	plunger pressure 5.5 bar			
0.0 to 1.0 minutes	Polymers			
1.0 to 2.0 minutes	Carbon black; ZnO; stearic acid, Naftolen			
2.0 to 3.0 minutes	Silica & silane; other constituents of the 1st step			
3.0 to 3.0 minutes	Clean			
3.0 to 5.0 minutes	Mix, with speed variation if necessary,			
	in order to reach the ejection temperature			
5.0 minutes	Discharge batch (batch temperature 140 °C to 150 °C) and distribute on roll:			
	Cut in and fold over 3 x on left, 3 x on right, turn over			
	3 x for narrow roll nip, 3 x for broad roll nip			
	Draw out a rolled sheet			
24 hours intermediate store so		_		
24 hours intermediate storage	at room temperature to step 2			
2 <sup>nd</sup> step	WORCK LEET - Land Cilian Land Co			
2 step	W&P GK 1.5E kneader, filling level 0.55,			
	80 rpm, flow temperature 80 °C,			
	plunger pressure 5.5 bar			
0.0 to 1.0 minutes	Plasticize batch from step 1			
1.0 to 2.0 minutes	Carbon black			
2.0 minutes	Aerate, clean			
2.0 to 4.0 minutes	Maintain batch temperature at 145° by speed variation			
4.0 minutes	Discharge batch (batch temperature 145 °C to 155 °C)			
	and distribute on roll:			
	Cut in and fold over 3 x on left, 3 x on right, turn over			
	3 x for narrow roll nip, 3 x for broad roll nip			
<del> </del>	Draw out a rolled sheet			
4 hours intermediate storage	at room temperature to step 3			
3 <sup>rd</sup> step	W&P GK 1.5E kneader, filling level 0.53,			
•	40 rpm, flow temperature 50 °C,			
	plunger pressure 5.5 bar			
0.0 to 2.0 minutes	Batch from step 2, accelerator, sulfur			
2.0 minutes	Discharge batch (batch temperature 90 °C to 110 °C)			
	and distribute on roll:			
	Cut in and fold over 3 x on left, 3 x on right, turn over			
	3 x for narrow roll nip, 3 x for broad roll nip			
	Draw out a rolled sheet			
12 hours intermediate storage				
beginning of the tests				

6. Technological rubber testing takes place according to the test methods presented in

Table C.

Table C: Test methods

Physical testing	Standard / Conditions		
ML 1+4, 100 °C, 3 <sup>rd</sup> step (ME)	DIN 53523/3 ISO 667		
Mooney scorch, 130 °C	DIN 53523/3 ISO 667		
Scorch time t <sub>5</sub> (minutes)			
Scorch time 135 (minutes)			
Tensile test on standard bar S 1, 23 °C	DIN 53504, ISO 37		
Tensile test (MPa)			
Elongation at break (%)			
DIE C N/mm)	ASTM D 624		
Shore A hardness, 23 °C (SH)	DIN 53 505		
Viscoelastic properties	DIN 53 513, ISO 2856 50 N preliminary force and 25 N amplitude force, temperature-stabilization time 5 minutes; recording of measured values after 30 s test time		
Complex modulus E* (MPa) Loss factor tan $\delta$ (-)			

7. Table D below presents the application-related data of the mixtures compounded and tested according to Tables A to C.

Table D: Results

		Example 4 <u>Uhrlandt et al</u>	Example 5 <u>Uhrlandt et al</u>	Example 1 of present invention
ML 1+4, 100 °C, 3 <sup>rd</sup> step	ME	37	39	39
Mooney scorch 130 °C; small rotor		)		
Scorch time ts	min	17.9	18.1	19.9
Scorch time t <sub>35</sub>	min	22.2	22.7	23.9
Vulcanization time; 150 °C	min	18	18	18
Tensile strength	MPa	22.7	22.3	22.7
Elongation at break	%	577	563	577
Die C; 100 °C	N/mm	52	55	75
Shore A hardness	SH	56	55	55
Viscoelastic properties				
E*, 0 °C	MPa	8.0	7.9	8.1
E•, 60 °C	MPa	5.6	5.6	5.6
tan 8,0 °C	-	0.234	0.234	0.234
tan δ, 60 °C	-	0.132	0.137	0.139

Application No. 10/523,029 Declaration Under 37 C.F.R. § 1.132

8. The compound containing the silica according to Example 1 of the present invention exhibits a very balanced profile of rubber values; among the properties of the raw mixture, the improved processing safety stands out in particular, as indicated by the prolonged scorch times. Whereas the reinforcement properties and also the viscoelastic properties correspond to the reference level or are slightly improved at comparable Shore A hardness, a distinct increase of high-temperature tearing resistance in the ASTM D 624 test

(Die C) can be observed. This performance improvement by 36% and 44% respectively is an

important criterion above all for high-performance, SUV and motor-truck tires.

9. The undersigned declares further that all statements made herein of his own knowledge are true and that all statements made on information and belief are believed to be

true; and further that these statements were made with the knowledge that willful false

statements and the like so made are punishable by fine or imprisonment, or both, under

Section 1001 of Title 18 of the United States Code and that such willful false statements may

jeopardize the validity of this application or any patent issuing thereon.

10. Further declarant saith not.

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